POLISHING METHODS FOR KCl

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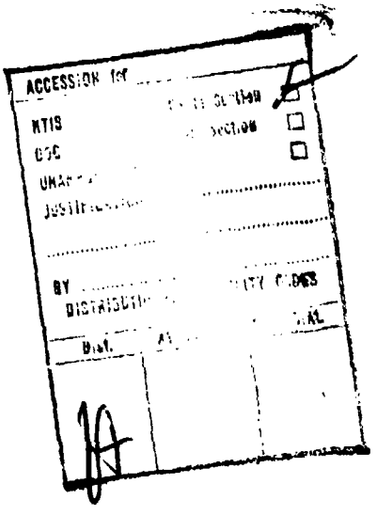

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13. ABSTRACT

Polishing techniques used by AFCRL for polishing alkali halides are described. A brief rationale is given for the techniques and materials required for low damage window polishing. Step-by-step procedures are outlined for use by technician or higher level individuals interested in window polishing. Particular attention is given to methods of rough cutting mechanical polishing and chemical polishing of KCl crystals.

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Preface

I am indebted to Morris Braunstein of Hughes Research Laboratories for many of the "black magic tricks" and generous discussions on this paper. The methods described are due to the efforts of M. Braunstein's group at Hughes Research Laboratories and Dr. J. W. Davisson of NRL.

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Polishing Methods for KCl

The following is a description of the salient features of the polishing methods used by AFCRL for KCl, and is offered as a guide for polishing KCl where optical flatness and parallelism are not critical. Extensions of these techniques can be suitably applied to the problem of a finished window.

Historically, the halide polishing problem began with the need for optical materials for early infrared spectroscopic instruments such as prisms and optical flats. Here the early problems of polishing were not critical, as surface absorption could not be easily detected in the early relatively impure materials. With todays purer materials (such as RAP grown KCl) showing bulk absorption near intrinsic values ($\beta \leq 1 \times 10^{-4}$ cm^{-1}), surface absorption is more easily detected and in many cases becomes the limiting factor in absorption coefficient measurements. In particular, with the intended application of halide materials as laser windows, the surface absorption has become a critical factor in the overall success of window development.

At this point in time (fall, 1973) many schemes have been tried to achieve super polish of not only KCl but other soft materials as well. There seems to be a black magic still associated with optical polishing in general and with soft materials in particular. It appears that many false starts have occurred, and worse, many unproven rumors have been initiated regarding what treatments can or cannot be used to produce low absorption surfaces on KCl (and other soft window materials).

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To dispel some of these thoughts, the following remark is offered: a gentle treatment is desirable with these materials. Certain handling techniques can indeed produce damage at unreasonable depths in these materials which can be best described as bulk damage. However, a certain amount of rough handling can be tolerated. Thus it appears that cleaving KCl can produce a certain amount of damage which exceeds say, a few microns in depth, but this can be polished off in normal polishing procedures.

There are presently two popular techniques for halide window polishing. The first is the method used by J. Davisson of NRL, which is a chemical polishing method. This, however, does not provide for the necessity of producing optically flat and parallel windows. Basically the method produces the most damage free surface, as no conventional mechanical polishing is used. To produce a window polish, only water polishing and chemical etching are used. Thus, the technique is in the strictest sense at present undeveloped for optical windows.

The second technique for polishing KCl is due to Hughes Research Laboratories. This method is more refined and approximates conventional polishing techniques, thus allowing fabrication of optically flat and parallel windows. By combining both methods we have the best of two worlds.

The following then is a description of the combined methods of Davisson and Braunstein.

I. ROUGH CUTTING

Several possibilities exist for rough cutting a window blank: To mention a few, diamond saw, string saw, cleaving (if appropriate) or even a hacksaw blade. Hughes has been using the string saw method with a high degree of success, and this is the recommended method. A diamond saw has been used at AFCRL, but due to vibration problems, this method is not recommended except when necessary. When this method is used, it is recommended that approximately 0.5 mm of material be removed from the cut (or cleaved) surface by water polishing.

After rough cutting, a rough polish may be placed on the sample using a minimum treatment on 600 grit SiC. A long polishing time is again not recommended. This step is done gently to flatten the starting surfaces.

For completeness a few words on water polishing are appropriate. The idea behind water polishing is controlled erosion of the material being polished. Water polishing is normally performed on a kitten ear wheel (essentially a fine velvet) using deionized water. For fast polishing, the wheel is turned at a fast speed and a constant drip of water is necessary. The water drip should be only enough to allow fresh water to freely dampen the wheel. Too much water will cause a wedge to

form or at best a very wavy surface. The final stages of water polishing are done on a stationary wheel which is only moderately damp. The final step should be a quick rinse with absolute ethanol or drying off of the sample to stop etching by any remaining water.

2. MECHANICAL POLISHING

Mechanical polishing of KCl is the most difficult step in the process of producing an acceptable sample surface. In the following only guidelines can be set up; experience is the best teacher. Several variations of the method described have been attempted, but the following has been the most successful.

The polishing wheel used at AFCRL is cast iron with a diameter between five and six times that of the sample to be polished. A pad of kitten ear is used as a cushion between the polishing cloth and the wheel. For optical polishing requiring precision flatness and parallelism, the pad is not used and a lapped wheel is required. (Hughes uses wheels to $\lambda/10$ flatness.)

The polishing cloth used is cotton flannel. At first it would seem that a smooth finish would preclude the use of flannel because of the nap. However, once the wheel is used a little, the nap will lie down and does not hinder the final flatness to any great extent. Ordinary white cotton flannel costing under a dollar a yard is sufficient.

The polishing agent is Linde B, 0.05μ Al_2O_3 in absolute ethanol (the good stuff). The solution is dilute as not very much Al_2O_3 is required for most polishing. As an added step in filtering Linde B, one can separate large particles by making a moderate solution of Linde B in ethanol and allowing it to settle for a few minutes. The smaller particles will remain in solution while the larger heavier particles will precipitate to the bottom of the container. One can then siphon off the supernatant and use this as the polishing solution.

The actual polishing procedure is easy to describe but does require patience in developing the knack of polishing such a soft material. The time involved in polishing a typical sample depends entirely on the smoothness of the starting surface, but a minimum of about 15 min per side is reasonable.

The polishing technique is conventional using the above wheel and solution. At AFCRL only surface quality is considered at this time so that the samples are treated much like metallurgical samples and polished by hand. The extension to precision optical polishing is left to the reader.

The wheel is turned at approximately 50 to 60 rpm and initially kept damp but not wet. The polishing solution is used on the wheel only once or twice during polishing, absolute ethanol being used after that to keep the wheel damp. When the sample has a reasonable finish, as judged by flatness and scratch free

appearance, the wheel is allowed to become nearly dry. As the wheel dries out, it will be noticed that the sample finish will become almost glass-like in appearance. Before this stage is reached the samples will cloud up when removed from the wheel. If this happens the wheel is merely too damp to finish, and a residue is left as the sample dries. During polishing, the pressure applied to the sample should not be too great. A noticeable drag from the wheel should be felt, however, too great a pressure will cause excessive damage, a texturing which is undesirable and finally imbedding of Al_2O_3 polishing compound.

The degree of polish attainable is almost a question of practice. A few comments on the philosophy of this technique may be useful. The use of cotton as a polishing cloth is very important. Cotton is very soft and does not tend to scratch the KCl in any significant way. The purposes of using small amounts of Al_2O_3 during polishing is only somewhat obvious. One of the features of using this dilute solution with a cotton cloth is that the Al_2O_3 will eventually become pressed into the polishing cloth yielding a much smaller exposed cutting edge. This means the scratches can be much smaller than .05μ at the end of the polishing procedure. It is also possible that continued use of the same polishing material will eventually wear these particles down to a smaller size much like the super polish technique of bowl feeding. That this particular phenomenon takes place is not clear since KCl is so soft.

At AFCRL, the samples are polished on both sides at one time. This is done by continually turning the sample over during the mechanical polishing. Also, the sample is rotated about its axis during polishing, giving a net-like polish mark structure. It is not clear that rotation is necessary, but during hand polishing it prevents a wedge formation on one edge of the sample.

As a final comment on this step several things should be noted: First, the sample is not washed after the final polish. There should be no significant Al_2O_3 on the surface, with only a small amount of buildup (if any) on the edges. Second, the determination of polish quality at any stage is not discussed here, as this is a matter of what is available. Nomarski (phase interference) microscopy is excellent, SEM work is also good - but the final judgement is again left up to the facility. During the polishing procedure, a small He - Ne laser can be used to estimate the rough polish by observing the degree of light scattering.

3. HEAT TREATMENT

Following the final mechanical polishing of KCl, a heat treatment has been shown to stabilize the sample surface. The nature of the heat treatment is trivial but extremely effective. A few words on its "raison d'etre".

During initial testing of optically polished KCl at Hughes Research Laboratories, it was found that samples were fogging rapidly between polishing and calorimetric measurements. Several methods of moisture proofing were attempted including rapid potting in carboline plastic. It was found that even with this method of passivation a certain amount of etching was present on removal of the sample from the carboline. The heat lamp process which follows was originally tried to keep the sample surface dry before potting. The result of this treatment seems to passivate the surface even without the carboline protective coating. By using this heat treatment as a last step, total absorption coefficients of KCl samples (RAP grown) are consistently attainable in the mid $10^{-4}cm^{-1}$ range (without chemical polishing). In addition, the samples thus treated are in fact more resistant to moisture and fogging than those which are untreated. As a final comment, it seems that the heating process has some annealing effect whose kinetic behavior is not well understood at this time.

The treatment may be performed in one of two ways, either using a small oven or a heat lamp. At AFCRL a heat lamp is used, but Hughes reports that the oven method is convenient for more than one sample and is equally effective. The oven method is easiest to describe—the sample is always placed in a small dry oven set at 50 to 60°C for a further period of not less than 15 min. If the sample is not to be subjected to further processing for some time, it may be left there for several days. If potting is desired, the sample is merely dipped while still warm (but not hot) into the self curing plastic. By wrapping a string around the sample before dipping, the sample may be unsealed much like a package of cigarettes. The sample may be stored for several months in this protective coating without severe degradation.

The heat lamp method if equally simple. The sample is placed under a small 250 W reflector type heat lamp. At AFCRL this arrangement is set up on a ring stand, and the sample is set on a watch glass to prevent rolling around during the treatment. The lamp is set about 3 to 4 in. from the sample. The sample is turned a number of times at first to prevent uneven heating. When the treatment is done (after something more than 15 min) it may be potted in the same fashion as described above. Please note that dipping a hot sample into a cold liquid will fracture the crystal.

4. CHEMICAL POLISHING

The chemical polishing method described here is merely a refinement of that developed by John Davisson at the Naval Research Laboratories. As described before, chemical polishing alone is not a developed technique for producing

optically flat surfaces. Thus, it is used in conjunction with mechanical polishing to achieve this end.

While chemical polishing does produce a surface smoothness which appears quite good, neither the rms flatness is known, nor has the polishing time been optimized. The principle feature of this polishing method is the damage-free and relatively passive surface it produces. Observe that one does pay for this, however, since this surface is so inactive that film adhesion becomes a serious problem.

The chemical polishing technique is best described in a step wise manner. The materials needed are:

(1) Reagent grade HCl,
(2) Absolute ethanol,
(3) A glass squeeze bottle,
(4) Freon 11 liquid,
(5) Four glass or glazed beakers, and
(6) Either platinum or nickel wire.

All work should be done under a hood. The solutions are set up in a line beginning with the HCl, then two beakers of ethanol followed by a beaker of fresh freon 11.

The nickel wire (or Pt) is used to hold the sample by merely wrapping it around the sample like a piece of string. The sample is polished by placing it in the HCl for from 20 sec to 1-1/2 min. The sample is gently agitated during this time to keep a constant flow of HCl on the surfaces. This prevents orange peeling to some extent. After this acid dip the sample is quickly rinsed in the first ethanol solution and is sprayed with ethanol on removal from this rinse. The sample is then again quickly sloshed in the third beaker (ethenol) and finally the Freon. On removal, the sample should dry spotless and clear.

. that the ethanol rinses should be short but complete. The reason for this is that ethanol will etch the surface if allowed to remain there. In addition, the final freon rinse should be changed every sample. Spotting occurs if this is not done.

Finally, the sample may be most easily released if the wire is cut (assuming nickel is used). The sample may be potted or coated at this point.

5. CONCLUSION

A practical description of polishing KCl has been described. The techniques have been proven in at least two separate laboratories as producing good quality optical finishes with low surface absorption.

The methods described are presented as guidelines for those laboratories involved in halide technology and reflect only the present state of the art. Actual hands-on technique is required as polishing is still an art more than a science and requires practice, patience and skill (and sometimes a little black magic).

6. OUTLINE OF PROCEDURE

a. Rough Cutting

String saw recommended. Flat surfaces are desirable before mechanical polish. Can use 600 SiC (or finer) if needed.

b. Mechanical Polish

Small wheel with cotton flannel cloth.
Al_2O_3 in absolute ethanol (Linde B - 0.05μ) dilute solution.

Wheel turned - 50-60 rpm.
Add Al_2O_3 only once or twice then ethanol to keep wheel damp.

Final polish on nearly dry wheel - don't rinse.
[USE ONLY GLASS CONTAINERS FOR POLISHING SOLUTIONS AND ETHANOL.]

c. Heat Treatment

(Recommended for all samples whether chemically polished or not.)
Heat lamp or oven for at least 15 minutes at 50 to 60°C.

d. Chemical Polishing

(1) Glass beakers under hood

(a) Reagent (concentrated) HCl,
(b) Absolute ethanol,
(c) Absolute ethanol,
(d) Freon 11 (liquid) fresh for each sample.

Use Ni or Pt wire to hold sample.

Note: Be sure sample is cool before starting.

(1) Dip in beaker 1 for from 30 seconds to 1-1/2 minutes.

Polycrystalline samples 30 seconds or less to prevent grain boundary etching; single crystal as above.

(2) Quick but thorough rinse beaker 2 followed by spray ethanol.

(3) Second quick ethanol rinse beaker 3.

(4) Thorough rinse in freon (beaker 4).